

# Microchemical Journal

*devoted to the application of microtechniques  
in all branches of science*

EDITOR-IN-CHIEF

*Al Steyermark*

VOLUME 15

1970

*Published under the auspices of the American Microchemical  
Society by*



ACADEMIC PRESS

*New York and London*

UNIVERSITY OF HAWAII LIBRARY

Copyright ©, 1970, by Academic Press, Inc.

ALL RIGHTS RESERVED

No part of this volume may be reproduced in any form,  
by photostat, microfilm, by retrieval system, or any other  
means, without written permission from the publishers.

MADE IN THE UNITED STATES OF AMERICA

# CONTENTS OF VOLUME 15

NUMBER 1, MARCH 1970

|                                                                                                                                                                  |     |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| J. MÉNDEZ. Ultraviolet Spectral Study of Indoles .....                                                                                                           | 1   |
| S. PANDE AND M. L. VERMA. Microdetermination of L-Leucine Separately and in a Mixture of Amino Acids .....                                                       | 6   |
| MOHAMED ZAKI BARAKAT, AHMED SHAWKI FAYZALLA, AND SHAKER TALKHAN EL AASSAR. The Microdetermination of Salicylic Acid and Certain Derivatives .....                | 10  |
| E. SAWICKI, C. R. SAWICKI, C. C. GOLDEN, AND T. KOBER. Fluorimetric and Colorimetric Methods of Analysis for Histamine .....                                     | 25  |
| O. C. SAXENA. Direct Titrimetric Determination of Samarium and Neodymium .....                                                                                   | 38  |
| R. BASSETTE, M. KUNDIGER, AND O. L. HANSON. Body Fluid Ketones Related to Fasting, Diabetes, and Tests of Blood Glucose Tolerance ...                            | 42  |
| JAY NEMATOLLAHI, WALLACE GUESS, AND JOHN AUTIAN. Pyrolytic Characterization of Some Plastics by a Modified Gas Chromatography .....                              | 53  |
| ZBIGNIEW GREGOROWICZ AND JÓSEF SLIWOK. Indexes for Estimation of Developing Reagents in Thin-Layer Chromatography .....                                          | 60  |
| A. K. SAXENA. Use of Microcosmic Salt as a new Titrant for the Microdetermination of Iodoacetic and Oxalic Acids .....                                           | 64  |
| LÁSZLÓ SZEKERES. New Data on the Determination of Calcium, Strontium and Barium Ions in the Presence of Each Other .....                                         | 66  |
| ANTHONY V. DETHOMAS AND MARK ZDANKIEWICZ, AND DAVID WITKOVIC. A Modified "Sandwich" Microapparatus for Descending Thin-Layer Chromatography .....                | 71  |
| ANTHONY V. DETHOMAS AND MARK ZDANKIEWICZ. A Simple Pre-equilibrium Apparatus for Thin-Layer Chromatography .....                                                 | 74  |
| S. CHANDRA AND K. L. YADAVA. Oxidation of Some Phenolic Compounds with Cu(III) .....                                                                             | 78  |
| JOSEPH F. ALICINO. Assay for Hydroxyurea .....                                                                                                                   | 83  |
| TAO WONG AND H. T. RUAN. Phosphocreatine. III. A Potential Error in the Determination of Phosphate Moiety of Phosphocreatine .....                               | 88  |
| P. C. SINHA AND O. C. SAXENA. Titrimetric Microdetermination of Beryllium .....                                                                                  | 92  |
| ALAN K. MUNSON, J. ROBERT MUELLER, AND MICHAEL YANNONE. An Inexpensive Sample Applicator for Thin-Layer Chromatography .....                                     | 95  |
| V. FANO AND M. SCALVINI. A Versatile Automatic Polarograph with Hanging Mercury Drop Electrode .....                                                             | 97  |
| BENJAMIN W. GRUNBAUM AND NELLO PACE. Microchemical Urinalysis. V. Quantitative Analysis of Individual Urinary 17-Ketosteroids by Thin-Layer Chromatography ..... | 103 |

# CONTENTS OF VOLUME 15

|                                                                                                                                                                 |     |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| P. K. JAISWAL. Oxidation of Some Carbohydrates with Ag(III) .....                                                                                               | 122 |
| PETER HAUX AND SAMUEL NATELSON. Identification of Renal Calculi on<br>Micro Samples by Infrared Analysis .....                                                  | 126 |
| RONALD H. LAESSIG. Simultaneous Automated Submicrodetermination of<br>Glucose, Blood Urea Nitrogen, and Uric Acid on 25 Microliters of<br>Capillary Blood ..... | 138 |
| BOOK REVIEWS .....                                                                                                                                              | 147 |

## NUMBER 2, JUNE 1970

|                                                                                                                                                                                            |     |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| W. B. BURTON. Synthesis of 2-Imidazolidinone- <sup>14</sup> C .....                                                                                                                        | 161 |
| A. K. SAXENA. Use of Microcosmic Salt as a New Titrant for the Micro-<br>determination of Acetic and Glycolic Acids .....                                                                  | 171 |
| K. F. SUGAWARA AND D. E. CAMPBELL. The Analysis of Binary and Ternary<br>Thin Alloy Films of Tantalum, Titanium, Niobium, Zirconium,<br>Molybdenum, and Beryllium .....                    | 173 |
| M. F. BARAKAT AND MAGDA ABDEL-GHANY. Differential Estimation of Low<br>Energy Beta-Emitters, <sup>3</sup> H, <sup>14</sup> C, and <sup>35</sup> S in Multilabeled Organic<br>Samples ..... | 184 |
| J. PAVEL, R. KUEBLER, AND H. WAGNER. Microdetermination of Fluorine in<br>Organic Compounds by Direct Measurement with a Fluoride Elec-<br>trode .....                                     | 192 |
| D. A. SHEARER AND G. F. MORRIS. Microdetermination of Fluorine in<br>Organic Compounds with a Fluoride Ion Electrode Following an<br>Oxygen Flask Combustion .....                         | 199 |
| P. K. JAISWAL. Oxidation of Ferrous Sulfate with Ag(III) .....                                                                                                                             | 205 |
| R. E. SCHAAD. Thin-Layer-Chromatographic Separation of Some Polynuclear<br>Hydrocarbons .....                                                                                              | 208 |
| SAFWAT W. BISHARA. Indirect Polarographic Method for the Microdeter-<br>mination of Sulfur in Organic Compounds .....                                                                      | 211 |
| Z. STEFANAC, Z. SLIPEČEVIĆ, AND Z. RAKOVIĆ-TRESIĆ. Direct Microdeter-<br>mination of Oxygen by Static Flash Combustion Pyrolysis .....                                                     | 218 |
| H. KHALIFA AND B. N. BARSOUM. Applications Involving the Iodide Ion.<br>V. Determination of Small Amounts of Platinum and Analysis of Its<br>Mixtures with Some Metal Ions .....           | 224 |
| MARTHA I. WALTERS AND H. W. GERARDE. An Ultramicromethod for the<br>Determination of Conjugated and Total Bilirubin in Serum or Plasma .....                                               | 231 |
| J. RAMÍREZ-MUÑOZ AND W. F. ULRICH. Relationship Between Sensitivity and<br>Precision in Atomic-Absorption Flame Photometry .....                                                           | 244 |

# CONTENTS OF VOLUME 15

|                                                                                                                                                                                                                                            |     |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| J. RAMÍREZ-MUÑOZ. Chemico-analytical Selectivity and Sensitivity in Atomic-Absorption Flame Photometry .....                                                                                                                               | 253 |
| J. RAMÍREZ-MUÑOZ. The Dilution Process in Sample Preparation for Atomic-Absorption Flame Photometry .....                                                                                                                                  | 271 |
| J. RAMÍREZ-MUÑOZ. The Concentration Process in Sample Preparation for Atomic-Absorption Flame Photometry .....                                                                                                                             | 277 |
| O. C. SAXENA. Microdetermination of Vitamin B <sub>6</sub> : Gold Chloride as Oxidizing Agent .....                                                                                                                                        | 281 |
| HARVEY W. YUROW AND SAMUEL SASS. Analytical Reactions in Trifluoroacetic Acid. II. Organic Spot Test Parameters .....                                                                                                                      | 285 |
| E. SAWICKI, T. W. WINFIELD, AND C. R. SAWICKI. Solvent Effects in Photometric Analysis .....                                                                                                                                               | 294 |
| KAN-ICHI NAKAMURA, KIKUSHIGE ONO, AND KATSURO KAWADA. Simultaneous Determination of Hydrogen, and Nitrogen in Organic Compounds by a Gas-Volumetric Method Using a Newly Designed Pt-P <sub>2</sub> O <sub>5</sub> Electrolytic Cell ..... | 364 |
| BOOK REVIEWS .....                                                                                                                                                                                                                         | 374 |

## NUMBER 3, SEPTEMBER 1970

|                                                                                                                                                                                                                                                  |     |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| VLASTIMIL REZL. Simultaneous Determination of Carbon, Hydrogen, and Nitrogen by Means of Gas Chromatography .....                                                                                                                                | 381 |
| O. C. SAXENA. Direct Titrimetric Microdetermination of L-Arginine. I. Direct Estimations of L-Arginine and DL-Valine, and L-Arginine and DL-Alanine; and L-Arginine, DL-Valine, and DL-Alanine Together in One Solution Without Separating ..... | 391 |
| MARILYN E. HOUSER AND MAE I. FAUTH. Indirect Determination of Nitrate, Nitrite, and Nitro Groups by Atomic Absorption Spectrophotometry ..                                                                                                       | 399 |
| G. E. SECOR AND L. M. WHITE. The Coulometric Microdetermination of Iodide After Oxygen Flask Combustion of Organic Compounds. The Use of Iodate, Biiodate, and Iodide as Primary Standards .....                                                 | 409 |
| H. KHALIFA AND Y. M. ISSA. Applications Involving the Iodide Ion. VI. Determination of Thallium (I) and Analysis of Its Mixtures with Some Metal Ions .....                                                                                      | 415 |
| V. FANO. Submicrogram Determination of Manganese with Other Elements by Polarography .....                                                                                                                                                       | 422 |
| HARVEY W. YUROW AND SAMUEL SASS. Detection of Various Alicyclic Compounds Via the Komarowsky Reaction .....                                                                                                                                      | 428 |
| P. K. JAISWAL. Tripositive Copper as a Titrant: Determination of Some Sugars .....                                                                                                                                                               | 434 |

## CONTENTS OF VOLUME 15

|                                                                                                                                                                            |     |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| FREDERICK A. ZYDECK. Determination of the Minimal Amount of Antigen Detected by the Electroprecipitin Test on Cellulose Acetate .....                                      | 438 |
| F. A. SORRENTINO, NANCY J. WITIAK, AND J. PAUL. Spectrophotometric Determination of Silicon in the Presence of Germanium .....                                             | 441 |
| F. A. SORRENTINO AND J. PAUL. Simultaneous Determination of Arsenic, Germanium, Phosphorus, and Silicon .....                                                              | 446 |
| WALTER SELIG. Semimicrodetermination of Oxalate with a Lead-Specific Electrode .....                                                                                       | 452 |
| A. K. SAXENA. Microdetermination of Glycolic Acid with Guanidine Carbonate as a Titrant .....                                                                              | 459 |
| KAN-ICHI NAKAMURA, MASA-AKI NISHIMURA, AND TETSUO MITSUI. Coulometric Microdetermination of Oxygen in Organic Compounds. ....                                              | 461 |
| S. S. M. HASSAN AND M. T. M. ZAKI. Microdetermination of the Hydrazine Function: A New Gasometric Method Based on Oxidation with Benzoquinone .....                        | 470 |
| E. J. POZIOMEK, A. F. FENTIMAN, AND R. H. POIRIER. A Microchemical Study of the Photodegradation of 4,4'-Bis(diethylamino)benzophenone Oxime on Silica Gel .....           | 475 |
| KEIICHIRO HOZUMI, OSAMU TSUJI, AND HIDEYA KUSHIMA. An Automatic Microdetermination of Carbon, Hydrogen, and Nitrogen in Organic Compounds Using Data Printing System ..... | 481 |
| BOOK REVIEWS .....                                                                                                                                                         | 498 |
| ANNOUNCEMENT .....                                                                                                                                                         | 508 |
| ERRATUM .....                                                                                                                                                              | 517 |

## NUMBER 4, DECEMBER 1970

|                                                                                                                                                                                                                      |     |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| JOHN T. STOCK AND ROBERT D. BRAUN. Dissimilar-Metal and "Titrant-Stream" Electrode Systems in the Potentiometric Titration of Millimolar Concentrations of Copper(II) in Dimethylformamide with Titanium (III) ..... | 519 |
| PAUL CARTER. Ultramicroestimation of Human Serum Albumin: Binding of the Cationic Dye, 5,5'-Dibromo-o-cresolsulfonphthalein .....                                                                                    | 531 |
| MRINAL K. DAS AND A. K. MAJUMDAR. Spectrophotometric Determination of Copper(II) with $\beta$ -Benzoyl- $\alpha$ -pyridyl Thiourea .....                                                                             | 540 |
| ZBIGNIEW GREGOROWICZ AND JÓZEF ŚLIWIÓK. Application of New Fuchsine for Quantitative Determination of Organic Substances in Thin-Layer Chromatography .....                                                          | 545 |
| VASANT L. SHAH AND SATENDRA P. SANGAL. Composition and Stability Constants of Cerium-Chromotrope 2R Chelate and the Spectrophotometric Determination of Cerium .....                                                 | 548 |

# CONTENTS OF VOLUME 15

|                                                                                                                                                                                                                                 |     |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| DONALD E. OKEN. Quantitation of Picogram Quantities of Serum Albumin by Ultramicrodisc Electrophoresis and Direct Densitometry .....                                                                                            | 557 |
| JOHN T. STOCK. Electrode Deactivation in the Anodic Voltammetry of Some Phenolic Derivatives of Isoquinoline .....                                                                                                              | 564 |
| B. L. GOYDISH. The Quantitative Determination of Boron in Glasses Used as Encapsulants for Electronic Devices .....                                                                                                             | 572 |
| M. EDRISSI, A MASSOUMI, AND J. A. W. DALZIEL. A Selective Differential Spectrophotometric Method for the Determination of Mercury(II) Using Tris(2-thiopyridine-1-oxide)-iron(III) as Reagent .....                             | 579 |
| G. S. R. KRISHNA MURTI, A. V. MOHARIR, AND V. A. K. SARMA. Spectrophotometric Determination of Iron with Orthophenanthroline .....                                                                                              | 585 |
| C. E. CHILDS AND E. B. HENNER. A Direct Comparison of the Pregl, Dumas, Perkin-Elmer, and Hewlett-Packard (F&M) Carbon-Hydrogen-Nitrogen Procedures .....                                                                       | 590 |
| J. A. HOWELL, G. E. LININGTON, AND D. F. BOLTZ. The Preparation and Indirect Spectrophotometric Determination of Total Oxidizing Capacity of Chlorine Dioxide in Acidic Solution .....                                          | 598 |
| JUI-CHANG WANG AND K. L. CHENG. Precipitation of Tellurium with Bis-muthiol II .....                                                                                                                                            | 607 |
| F. BERMEJO-MARTINEZ AND J. A. RODRIGUEZ CAMPOS. Analytical Applications of Chelons. LVII. Chelometric Determination of Bismuth with DTPA and Reciprocally .....                                                                 | 622 |
| K. GENEST AND LORNA J. LOWRY. Microcrystalloptic Tests for Some Lupine and Ormosia Alkaloids .....                                                                                                                              | 625 |
| JOSEPH K. SAMUELS III AND D. F. BOLTZ. Near-Infrared Spectrophotometric Determination of Germanium by Modified Heteropoly Blue Method ..                                                                                        | 638 |
| K. N. JOHRI AND HARISH C. MEHRA. Trace Metal Analysis by Combined Thin-Layer Chromatography Incorporating Fluorescent Support and Ring Oven Colorimetry .....                                                                   | 642 |
| K. N. JOHRI, HARISH C. MEHRA, AND N. K. KAUSHIK. Suggested Procedure for Microdetermination of Arsenic in Arsenical Animal Feed .....                                                                                           | 649 |
| H. FLASCHKA AND R. WEISS. The Extraction and Photometric Determination of Zinc in the Presence of Large Amounts of Lead (Mercury, Copper, or Silver) Using 1-(2-Pyridylazo)-2-naphthol (PAN) and Employing Iodide Masking ..... | 653 |
| BENJAMIN W. GRUNBAUM AND NELLO PACE. Microchemical Urinalysis. VI. Determination of Sodium, Potassium, Calcium, and Magnesium by Atomic Absorption Spectrophotometry in the Microliter Range of Urine. ....                     | 666 |
| BENJAMIN W. GRUNBAUM AND NELLO PACE. Microchemical Urinalysis. VII. Determination of Citric Acid in Microliter Quantities of Urine ..                                                                                           | 673 |

## CONTENTS OF VOLUME 15

|                                                                                                                 |     |
|-----------------------------------------------------------------------------------------------------------------|-----|
| DAVID B. SABINE. Photomicrographs of Metallo Amino Acid Complexes ..                                            | 677 |
| BENJAMIN W. GRUNBAUM. Reliability of the Grunbaum Pipet as a Self-Cleaning Device .....                         | 680 |
| GEOFFREY GORDON AND GEORGE M. MALACINSKI. An Improved Microinjection Apparatus for Biochemical Embryology ..... | 685 |
| BOOK REVIEWS .....                                                                                              | 692 |
| AUTHOR INDEX .....                                                                                              | 698 |